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RUBBER RESEARCH AND TECHNOLOGY AT THE NATIONAL BUREAU OF STANDARDS

By LAWRENCE A. WOOD

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By Lawrence A. Wood

ABSTRACT

This paper consists of a summary of the activities of the National Bureau of Standards relating to natural rubber, synthetic rubbers, and related materials. It gives a brief outline of the work carried out in each of 13 different fields of investigation, and concludes with 174 references to publications by members of the staff of the National Bureau of Standards.

I. INTRODUCTION

About 4 years after its establishment in 1901, the National Bureau of Standards began its first work on rubber. The first problem was the development of methods of analysis of cable insulation. Over the years since then a great many different types of investigation have been conducted.

This diversified scientific research on rubber has led to the solution of many problems of interest to both the Government and private industry. Much of the research has been concerned with the determination of fundamental properties and constants, and the development of methods of test. In addition, the National Bureau of Standards makes studies of the over-all performance of rubber products, including tires, assists in the development of specifications for synthetic rubbers and for rubber products of all kinds, and gives technical assistance and advice to other government agencies and the public.

The nature and emphasis of the work have changed, over the years, corresponding to the accumulation of knowledge and to the great expansion of the rubber industry. For example, much of the work before 1925 concerned chemical analysis and physical testing of natural rubber. After 1940 this type of work again came into prominence in connection with the synthetic rubbers. In the intervening years much attention was given to the determination of physical constants and properties. Work on wild rubbers, largely given up about 1915, had to be resumed between 1940 and 1945 when supplies of plantation rubber from the Far East were cut off. The national synthetic rubber program has called for extensive work since 1940 on the monomers and other raw materials used in the production of the synthetic rubbers.

The rubber industry has adopted a great many testing devices first developed at the National Bureau of Standards. Among the more outstanding may be mentioned the endurance wheel for tire testing and the abrasion test machine. Likewise, its research work has fur-

nished the basis for many standard practices now employed in industrial testing. Examples of this are the use of flat dumbbell test specimens and the introduction of standard conditions of temperature and humidity. More complete details regarding these matters are given below in the section dealing with physical testing.

International contacts were established with a number of European rubber laboratories in 1938, when two members of the Rubber Section attended the International Rubber Technology Conference in London. They investigated the research activities of a number of laboratories in England, France, and Holland as well as the major German synthetic rubber research laboratory at Leverkusen. During 1943 and 1944 a member of the Section was detailed to Belem, Para, Brazil, to establish a rubber testing and research laboratory for the Brazilian government as a part of the Instituto Agronomico do Norte, a national agricultural research institute. The Rubber Section has continued to act in an advisory capacity in this laboratory, which has recently taken over the operation of the Ford plantations in Brazil. In the Fall of 1946 a British physicist from the British Rubber Producers' Research Association laboratories was detailed to spend a year as guest worker at the National Bureau of Standards.

Early in 1943 the Office of the Rubber Director requested the National Bureau of Standards to expand its work on synthetic rubber and to assist the Rubber Reserve Co. in the standardization of the quality of the synthetic rubber. Since then it has participated in the program to the full extent of its facilities. Supplementing the facilities of the Bureau's own grounds where most of the work is done, there is also a laboratory at 203 Bryant Street, N. W., in Washington, for chemical analyses of synthetic rubbers.

The National Bureau of Standards has played a considerable part in developing a number of the tests, described more fully in a later section, now being used in the synthetic rubber plants. Among them are the use of refractive index as a method of determining the styrene content of a GR–S copolymer and the determination of freezing points as a method of measuring the purity of styrene, butadiene, and other monomers.

The scope of the research at the National Bureau of Standards, it will be noted, does not overlap the work of the Department of Agriculture, which deals with the studies relating to the cultivation and preparation of rubber from different botanical sources. Furthermore, it does not duplicate the work of other government agencies, such as the military services, which are usually interested in the testing of finished rubber products of specific concern only to the particular agency.

An extensive program of testing and research on plastics supplements the work on rubber in many respects. There is particularly close coordination in the fields of polymerization, determination of molecular weights, and similar phases.

At the present time about 45 persons at the National Bureau of Standards spend their full time in work on natural and synthetic rubbers and materials used in the synthetic rubber program. In addition, there are a number of others devoting part time or acting in a supervisory capacity. The work at present is financed in part by

direct Congressional appropriations and in part by a transfer of funds from the Office of Rubber Reserve. Some testing work is supported by transfer of funds from other agencies.

II. RUBBER RESEARCH AND TECHNOLOGY

The possible number of phases of research on rubber is relatively large, and profitable investigations may be carried out in many different fields. This section lists 13 of these fields and shows why work is required in each of them. It also outlines very briefly what the National Bureau of Standards has done on each subject in the past.

Where the work is of limited interest, the results are presented in the form of reports to the specific organization concerned. Where the work is of general interest, the results are presented in the form of printed publications. Over the years the research investigations on rubbers and related materials at the National Bureau of Standards have led to the publication of over 170 papers by members of its staff. In this section, references are made, by numbers in brackets, to a list of these publications, classified under the same 13 headings given in section III.

1. NATURAL RUBBER: CRUDE-RUBBER PRODUCTION, EVALUATION, AND TESTING

The system under which natural rubber was bought by American manufacturers before the war was such as to give them almost no control over the quality of the rubber they received. The uniformity of day-to-day shipments was so poor that each manufacturer was required to carry on large-scale blending operations to maintain moderately constant properties. Experience with synthetic rubbers, having a uniformity maintained by testing both at the producing plant and at the consuming factory, has led rubber product manufacturers to call for the development of new and improved test methods for crude natural rubber. These must be methods that can be applied both on the plantations, where virtually no testing has been done hitherto, and also in the American rubber factories.

Between 1942 and 1945 the National Bureau of Standards tested more than 1000 samples of wild natural rubbers for the Rubber Development Corporation. On the basis of these tests a method of evaluating such rubbers has been proposed. Work is under way for the extension of this method to the evaluation of plantation rubbers.

A rubber laboratory [1 to 4] [1] was set up and operated under the direction of a member of the Rubber Section at the Instituto Agronomico do Norte in Belem, Para, Brazil, in 1942–44. As a result, the section has personnel with first-hand tropical experience in problems of natural rubber production and is continuing work on some of these problems.

2. SYNTHETIC RUBBERS: PRODUCTION AND TESTING

America's war-time synthetic rubber program affords a remarkably close parallel to this country's work on the atomic bomb. On each program the total expenditures were of the order of two billion dollars, the rubber program producing some counterbalancing income. Each

[1] Figures in brackets indicate the literature references at the end of this paper.

was strikingly successful and was completed according to a schedule laid out several years in advance. In each there was unprecedented teamwork by large groups of men working as parts of many different organizations under Government direction and control. Finally, and most important from the standpoint of the present discussion, each program was an engineering achievement based entirely on fundamental knowledge gained before the war. Much subsidiary research had to be done, but the basic principles in each case were completely familiar to scientists in 1939. It is significant that no new variety of synthetic rubber of demonstrated value was discovered during the war.

Engineering accomplishments in synthetic rubber have by now taken full advantage of the known principles of the fundamental science involved. It is generally agreed that future progress depends on developing new basic principles. We can no longer rely on German laboratories, where so many of these principles have been developed in the past. There is obvious need for fundamental research on the production of synthetic rubbers.

As new types of synthetic rubber are made in the laboratory, better methods should be developed to evaluate their qualities. At present the complete evaluation is highly empirical and requires extensive service tests. The limited quantity of material available precludes such tests on more than a very small fraction of the polymers produced. After a rubber has gone into large-scale production, methods must be available to test it for uniformity.

A visit by two of the members of the Rubber Section to the major German synthetic rubber research laboratory at Leverkusen in 1938, and a thorough survey of the published literature led to the issuance of NBS Circular C427 [6] on synthetic rubber in June 1940. The Circular, which appeared at a very opportune time, has had a wide distribution, and was much used in the early days of the American synthetic rubber program.

Since 1943, when the work on synthetic rubber was expanded, more than 100 extensive mimeographed reports on this work have been written and distributed through the Office of Rubber Reserve to participants in the national rubber program. Because they have not as yet been released for general distribution, they are not listed with the publications in section III.

The test methods now in use in the American synthetic rubber plants are so many and varied that several large laboratory manuals have been issued solely to describe the procedures. They have been developed by a cooperative effort on the part of many different laboratories, coordinated by the Office of Rubber Reserve. The National Bureau of Standards has contributed to the development of a number of these methods and to the committee work involved in their general adoption. A member of the Bureau staff was selected for the Committee on Specifications for Synthetic Rubbers, and has served as chairman of its Subcommittee on Test Methods. Others have participated in the activities of the Polymer Research Discussion Group.

GR-S, the synthetic rubber being produced in largest quantities, is a copolymer of butadiene and styrene. Consequently, a determina-

tion of the ratio of the two constituents of the polymer is an important analytical problem. The method now in use in the plants producing GR-S was developed at the National Bureau of Standards in 1944. It involves a measurement of the refractive index of the rubber, from which impurities have been removed by extraction. The relation used is based on high-precision measurements of the carbon-hydrogen ratio in GR-S. The reproducibility of the index measurement is such that repeated determinations of the styrene content rarely differ by more than 0.2 percent in the normal GR-S, which contains about 24 percent of bound styrene.

3. LATEX

Rubber emerges from the trunk of a rubber tree, or from the polymerization reactor in synthetic rubber production, in the form of liquid latex, similar in appearance to milk. More and more rubber products each year are made directly from latex, especially such articles as rubber gloves, rubber thread, waterproofed fabrics, and sponge-rubber upholstery. The storage, transportation, and use of latex give rise to problems of a specialized nature.

The work on latex at the National Bureau of Standards has been limited largely to the compiling of general information on the subject [9]. Inquiries from small business firms are particularly frequent because many latex products can be readily produced on a small scale without expensive equipment.

4. PURIFICATION OF RUBBER

Crude rubber, either natural or synthetic, usually contains between 5 and 10 percent of nonrubber constituents. Removal of these constituents, at least partially, is a necessary preliminary to a great many different types of fundamental studies of the properties of rubbers. Several important methods for the purification of natural rubber have been developed [10 to 13].

According to one of these methods [13] crude rubber or latex is digested with water under pressure at 190° C to hydrolyze the proteins, yielding products which are then removed by extraction, together with the resins and other impurities. The purified material contains about 99.5 percent of rubber hydrocarbon. Rubber purified in this manner was used in investigations of crystallization [24], specific heat [41], specific volume and expansivity [42], heats of combustion [43], electrical properties [48, 49], refractive index [57], photoelasticity [58], and heats of reaction [70].

A different method of purification, calling for digestion of the proteins in latex with the enzyme trypsin, has also been developed [12]. Rubber purified by this method has been used in investigations [11, 12] of gel content, crystallization, and carbon-hydrogen ratio, as well as in studies of the stress-strain behavior of sol and gel rubbers [33], X-ray diffraction [59 to 61], crystallization [28], and molecular distillation [10].

Distillation is an important method for the separation and purification of different liquids. An attempt has been made to perform a molecular distillation of the rubber hydrocarbon [10]. The results were somewhat inconclusive.

Synthetic rubbers have been purified by solution in benzene and precipitation with alcohol. Other methods of preparation of pure synthetic rubbers are currently under investigation in order to obtain suitable materials for use in measurements of electrical properties, infrared absorption, and a number of related properties, as well as in determinations of the carbon-hydrogen ratio and the nature of the pyrolytic decomposition products.

5. FORMS OF RUBBER: CRYSTALLIZATION AND OTHER TRANSITIONS IN RUBBER

When natural rubber or some varieties of synthetic rubber are stretched, tiny crystals are formed in the rubber, which have an important effect on its mechanical properties. The complete absence of such crystals in GR–S synthetic rubber is probably responsible for its relatively low tensile strength. Studies of the formation of crystals in rubber at low temperatures are necessary for an understanding of its behavior in use at such temperatures. Many low-temperature laboratory tests, set up on an arbitrary basis before the significance of crystallization was realized, proved quite valueless in predicting the behavior of rubber products exposed to low temperatures over long periods of time. The fundamental work on crystallization [14 to 25] conducted over a 10-year period at the National Bureau of Standards has recently been having a significant bearing in setting up proper conditions of time and temperature for low-temperature testing.

Besides crystallization, there is another transition evident in natural rubber at about −70° C. At this transition temperature there is no abrupt change of volume or heat content, such as occurs during crystallization, but instead the rate of change of these properties with temperature undergoes a large change. For this reason it is called a second-order transition in contrast with a first-order transition, such as crystallization, in which the volume and heat content do undergo abrupt changes. All rubbers are found to possess second-order transitions, and to be inextensible at temperatures below that of the second-order transition. The results of fundamental research [24, 41, 42] have been of important assistance in understanding the nature of this transition, and from this understanding it has been possible to explain many effects connected with brittleness and other mechanical properties not only at low temperatures but also at room temperatures.

6. CONSTANTS AND PROPERTIES OF RUBBER

Investigations of the properties and constants of rubber have been under way at the National Bureau of Standards for many years. These constants and properties, once determined, have been widely used by the rubber industry. A compilation [26] of the values of the physical constants of natural rubber, largely determined at the Bureau, was presented at the International Conference on Rubber Technology held in London in 1938. Much work still remains to be done in determining similar values for the different synthetic rubbers [6].

Density and Specific Gravity.—The manufacturer of a rubber product buys his crude rubber on a weight basis at so much per pound. The product itself, however, is usually produced in a particular size,

occupying a required volume rather than having a required weight. Consequently, rubber manufacturers are very much interested in knowing exactly the relation of weight to volume, or in other words, in having accurate values of the density or specific gravity of the rubbers. The National Bureau of Standards has made determinations of values of these properties [27, 42, 51], which have found frequent application in industry.

Mechanical Properties.—The unique property by which one recognizes a rubber is its high extensibility followed by rapid and virtually complete retraction. Only recently has there been an approach to understanding the molecular mechanism by which this occurs. Considerable research on the mechanical properties of rubber has been required to furnish the basic data for the new theories. The results show that the extension of rubber is an uncoiling of long-chain molecules, and that the retractive force arises from thermal motions. The origin of the retractive force is thus analogous to the origin of the pressure in a gas in that it also arises from the thermal motion of the molecules. Obviously, an understanding of the origin of the elasticity is necessary in predicting which of the thousands of possible polymers will be extensible synthetic rubbers.

Recent work [29, 30] on the change of stiffness of natural and synthetic rubbers with change of temperature has furnished information with applications in this important field. The work also assists in devising the most suitable type of mechanical tests to apply in the routine testing of rubber. Work on the frictional properties of rubber [31], in addition to its obvious application to tires, has also served as a guide for other work on the slipperiness of flooring [35].

Thermal and Thermodynamic Properties.—Thermodynamic studies [41] initiated about 1931 yield information from which predictions may be made regarding the various chemical reactions in which different rubbers participate, including a determination of the most favorable conditions for the reactions. A summary [40] of the earlier work on the application of thermodynamics to the chemistry of natural rubber was presented before the International Rubber Technology Conference in London in 1938. More recent work has dealt with the different synthetic rubbers [37, 38].

Electrical Properties.—The wide use of rubber-insulated wire gives rise to the need for information on the electrical properties of rubber over a wide range of conditions of temperature, voltage, and frequency. The designing of rubber compounds to give specified electrical properties is a field of considerable importance to the industry and to the government, which is a large purchaser.

The National Bureau of Standards conducted an extensive research program [46 to 53] on the electrical properties of natural rubber between 1920 and 1940. The considerable advances made in understanding these properties led directly to greatly improved compounds for electrical insulation in wires and cables and to improved methods of test [47, 49].

More recent work has led to the development of a synthetic rubber compound suitable for undersea cables [45]. This compound and later variations of it found extensive use during the war.

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Optical Properties.—It is a generally unrecognized fact that light can be transmitted by rubber in thicknesses up to about one inch. Many familiar products contain fillers which are responsible for the opacity generally noted. Studies of the optical properties of rubber [54] yield important information about the molecular structure and are the basis for several analytical methods. For example, prewar work on the optical properties of natural rubber [57] pointed the way to the development of the method curently in use in the synthetic rubber plants for determining the bound styrene content of GR–S synthetic rubber, as described above in the section on synthetic rubbers.

X-ray Diffraction.—X-rays furnish a tool by which one can gain information about the arrangements of the atoms in rubber molecules and about the nature of crystallization [59 to 61]. During the war X-ray patterns were used to identify natural rubber in samples which had been submitted as supposed examples of a synthetic rubber. Some excellent new types of equipment for this work are now available.

Permeability to Gases.—Rubber is used to confine a gas in airships, meteorological balloons, inflatable life rafts, inner tubes for pneumatic tires, and many similar devices. As early as 1917, investigations were begun on the permeability of rubber to different gases [62 to 65].

Properties of Rubber in Solution.—Important information regarding the molecular weight and structural properties of a rubber is obtained from studies of its behavior in solution [66 to 68]. The work has a directly practical application to rubber cements.

7. CHEMICAL REACTIONS OF RUBBER

Vulcanization of Rubber.—The nature of the vulcanization reaction by which rubber is converted from a soft plastic material into a product of commercial utility is of the highest theoretical and practical significance. This reaction, which is the major change in the material as it passes through a rubber factory, was discovered more than a hundred years ago. Nevertheless, the nature and mechanism of the reaction are very little understood. The theories which have been proposed are partial and contradictory. Further progress calls for the application of new methods in a systematic study of this field. A few fundamental experimental studies [69 to 71] have been undertaken in the past. The advent of synthetic rubbers opens up many new possible approaches to this problem.

Aging.—Rubber articles do not usually retain their utility indefinitely. Laboratory research has demonstrated that the deterioration is an oxidation caused by the oxygen and ozone of the air. Likewise, research has developed antioxidants that increase the useful life of rubber products manyfold. With such products it is necessary to devise greatly accelerated aging tests in order to have results available in less time than the period of years required for natural aging to produce appreciable deterioration. A long-term testing program made it possible to investigate the relation of natural aging to accelerated aging [72 to 75].

Effect of Elevated Temperatures on Rubber.—A serious limitation in the applications of natural rubber and the synthetic rubbers (with the exception of the newly developed silicone rubbers) is the fact that they cannot be used at elevated temperatures. In use at temperatures

much above boiling water, their properties are no longer satisfactory and deterioration is relatively rapid. In an attempt to understand why this is so, studies have been made of the evolution of hydrogen sulfide from rubber at high temperatures [76, 77]. The studies also furnish information regarding the nature of the vulcanization reaction.

8. CONSTANTS AND PROPERTIES OF MONOMERS AND OTHER MATERIALS RELATED TO RUBBER

Synthetic rubbers are made by the process of polymerization, which transforms monomers made up of molecules containing not more than a few dozen atoms into polymers with molecules each containing thousands of atoms. The properties and constants of the liquid monomers used in synthetic rubber production are consequently of considerable interest and importance. In addition, detailed information is required on the compounds used in the production of the monomers and the impurities in them. Both the measurement of these properties and constants, and the compilation of extensive tables presenting the results in most applicable form, require highly specialized equipment, personnel, and experience. At the beginning of the war the National Bureau of Standards was in a unique position to render service to the synthetic rubber program, which required the determination of constants and properties of the monomers and other raw materials. A summary [79] of preliminary values of many of the physical properties of butadiene and styrene was issued by the National Bureau of Standards in 1942, while the synthetic rubber plants were still being designed and constructed. Work of this sort previously done on isoprene [84, 102, 103], the monomer related to natural rubber, was extended to the materials related to the synthetics. Thermal and thermodynamic properties [85 to 101] were stressed especially, and the results furnish useful information regarding reactions and most favorable conditions for production of the raw materials and for their polymerization to form the corresponding rubbers.

An extensive program of work on the collection, analysis, calculation, and compilation of data on the properties of hydrocarbons was begun at the National Bureau of Standards in 1942 as Project 44 of the American Petroleum Institute. This has resulted in the issuance of about 400 pages of tables [78] presenting selected self-consistent "best" values of properties, and in the issuance of almost 700 graphs showing infrared and ultraviolet spectra. As different sections of these tables and spectrograms have been issued they have been distributed by the Office of Rubber Reserve to those concerned with the production of synthetic rubber. They were declassified at the beginning of 1946 and made generally available to the public.

Analytical Methods.—The purity of butadiene, styrene, and other monomers used in synthetic rubber production is of very great importance in determining the rate of polymerization. Consequently, extensive development work has been done at the Bureau on analytical methods for these materials. Much attention, for example, has been given to the determination of freezing points as a method of measuring the purity of these liquids. This method [80] is one of the most satisfactory of those in use for determining the purity, but requires

considerable care and precision. In the case of styrene the freezing-point method is the one used in routine testing in the plants. With butadiene it is used as a referee method in cases of doubt.

The mass spectrometer has only recently been applied to the analysis of liquids and gases. It is particularly suitable for the hydrocarbons and related materials used in the production of the synthetic rubbers. The National Bureau of Standards has had one of the few instruments of this type that could be largely devoted to the synthetic rubber program [81]. The analyses often involve mixtures too complex for investigation by any other means.

Staff members of the National Bureau of Standards have served on the committees of the Office of Rubber Reserve, which were responsible for establishing specifications and test methods for butadiene, styrene, and related materials.

Densities and Other Simple Physical Properties.—The National Bureau of Standards has had long experience in making accurate measurements of densities and in compiling tables of physical properties of hydrocarbons, which have become standards in the petroleum industry. At the beginning of the synthetic rubber program there was need for such data and tables on butadiene and other hydrocarbons related to the production of synthetic rubber. It was, therefore, logical for the Rubber Reserve Co. to request the preparation of a standard table [83] of values of densities of the 11 hydrocarbons most important in the production of butadiene and another table [82] of the volume correction factors of mixtures of these hydrocarbons. These tables have been utilized very extensively by the butadiene producers and the operators of the polymerization plants.

Thermodynamic Properties.—The thermodynamic properties of monomers and related materials are important in the production of synthetic rubber for ascertaining the behavior of these materials under the different conditions of pressure and temperature that arise in the course of production and use. Of even greater importance perhaps is the fact that they also yield useful information regarding the extent of their chemical reactions and the prediction of the most favorable conditions for conducting these reactions. The reactions include both those utilized in the production of the monomers from the raw materials and those utilized in the polymerization of the monomers to produce the rubbers. The calculation of thermodynamic properties requires accurate experimental data on specific heats and heats of combustion.

The National Bureau of Standards, which had already obtained very reliable data on thermodynamic properties of different materials before the war, has recently directed special efforts to the materials important in the synthetic rubber program. The work on butadiene [86, 91, 95, 98, 100], for example, is one of the most complete investigations of thermal and thermodynamic properties ever made on a hydrocarbon. Materials important in the production of butadiene are the butenes [85, 88, 90, 91, 98, 101], the acetylenes [94], and the paraffins [96, 97]. Isobutene, one of the butenes studied, is also the major component of GR–I synthetic rubber, often called Butyl rubber. Isoprene [84, 102, 103], the monomer related to natural rubber, also appears as the minor component of GR–I.

The thermodynamic properties of styrene, the minor component of GR–S synthetic rubber, have also been studied [98 to 100]. In addition, similar studies have been made on materials important in the production of styrene, namely ethylene [86, 88, 90], benzene [87, 99], and the alkylbenzenes [87, 89, 99], especially ethylbenzene [92, 93, 98]. The tables [78] compiled as part of Research Project 44 of the American Petroleum Institute already mentioned, contain extensive data on thermodynamic properties.

9. CHEMICAL ANALYSIS OF RUBBER

Before 1920, chemical analysis was widely used to ensure that low-cost adulterants were not used in rubber products [104 to 121]. For many general purposes it was later supplanted by performance requirements and physical tests, but was retained where the composition, rather than the physical behavior, was of importance. Chemical analysis of raw synthetic rubber has been of assistance in maintaining uniformity of production. The National Bureau of Standards has developed and investigated a number of new methods for the Office of Rubber Reserve. Chemical analysis has also come into recent use in evaluating raw natural rubber and in determining relative amounts of natural and synthetic rubbers.

10. PHYSICAL TESTING OF RUBBER

As the most important property of a rubber is usually its ability to stretch and retract, the tests most commonly made on rubber are tests of its tensile properties. The quality of a rubber product is often judged by measurements of tensile strength, ultimate elongation, and the force required for a given elongation.

A great many of the methods and principles now used in American industrial laboratories for the physical testing of rubber originated in research at the National Bureau of Standards many years ago.

The first principles were laid down in early papers [136 to 138] presented before international congresses and developed more fully in NBS Circular C38, The Testing of Rubber Goods [134], of which five editions were required, beginning in 1912. Largely on the basis of investigations described in this publication, American practice has been to use flat dumbbell specimens instead of the ring specimens favored by the Europeans. From 1927–29 the Rubber Manufacturers Association, the Rubber Division of the American Chemical Society, and a number of rubber companies jointly supported work at the National Bureau of Standards that led to the formulation of recommended procedures and practices [131 to 133] in physical testing now used almost universally in this country. A summary of modern practices in the physical testing of rubber was written by a member of the Rubber Section as a portion of the French Encyclopedia of Rubber Technology [125].

Additional work in this field became necessary with the advent of synthetic rubber. In the first place, redeterminations of temperature coefficients, standard deviations of testing, and other quantities were required by the new materials. In the second place, closer control of certain variables in physical testing was found to be necessary

in controlling the production of a uniform synthetic rubber. Finally, the very fact that 15 Government-owned plants were required to produce rubber to meet the same specifications called for testing of higher accuracy than had heretofore been necessary. Interlaboratory cross-tests are being made regularly. As a result of the standardization of procedures, in which the National Bureau of Standards has taken a leading part, the precision and accuracy of rubber testing in this country have been notably improved.

Prior to 1942 the rubber industry could agree on no single test for determining the plastic properties of unvulcanized natural rubber. In order to control the production of synthetic rubber, it was essential to have a reliable method for measuring this property. The Rubber Reserve Co. and its agents, the companies operating the synthetic rubber plants, decided to employ the Mooney viscometer for this purpose. However, as manufactured at that time, it did not permit the reproducibility required to control the production of a uniform synthetic rubber. Responsibility for improvements in the design of the viscometer and for the standardization of test procedures was delegated to the National Bureau of Standards. As a result of these investigations, a viscometer of improved design was developed and is currently being manufactured. It has now become the most common means of measuring plastic properties in the rubber industry. Recommended procedures and factors affecting the results obtained in measurements with this viscometer have been described in a recent publication [122].

Among other publications relating to physical testing may be noted an abrasion test machine [130] in rather widespread use, a compression cutting test [126], and a study of the use of the Shore Durometer [123].

11. RUBBER PRODUCTS

Tires.—There are no completely satisfactory methods of predicting the life of tires under service conditions. Each of the larger tire manufacturers maintains test fleets and conducts road tests, which are notably expensive, time-consuming, and lacking in reproducibility. There is an urgent need for the development of methods by which the different factors in tire failure can be isolated and tested separately.

The endurance-test wheel for the testing of tires in the laboratory was pioneered at the National Bureau of Standards many years ago [144] and has been generally adopted by industry. Tires are run to failure under abnormally severe conditions of load, inflation, speed, and temperature. The type of failure desired can often be predetermined by the conditions of the test. The use of this test has screened out many defective compounds and types of construction, and reduced very considerably the amount of road testing necessary. Research on synthetic rubber tires has led to the development of a new method [139] for measuring tread wear during road tests by repeated weighing of the tire and wheel, and to a method of measuring internal temperatures in tires.

Reclaimed Rubber.—In normal times about one-third of the rubber in manufactured products in this country is reclaimed and used again. The processes used were developed by the industry

in the early 1900's and have not been notably altered since then. It seems likely that systematic research yielding a better understanding of the nature of the process would lead to new and improved methods. The reclaiming of synthetic rubbers has recently become a subject of importance. Work at the National Bureau of Standards in this important field has been limited for the most part to compilation of published information [149]. Although more than 15 years old, this publication is still in considerable demand, since it is almost the only printed source of specific information about reclaiming practices.

Coated Fabrics and Thin Films.—During both World Wars the military services called upon the National Bureau of Standards for special work on balloons, pontoons, liferafts, waterproofed goods, and similar rubber products. The work has continued steadily through the intervening years [150 to 153].

Brake Lining.—Varying amounts of rubber are contained in different types of brake linings. Methods of test and a special testing machine [155] have been developed for evaluating brake lining, especially for Government purchase.

Other Rubber Products.—Work on other rubber products has included investigations on such varied items as denture rubbers [159], garden hose [167], sponge rubber [164], jar rings used in canning [158], floor covering [160, 165], binders for foundry cores [166], and rubber cements [162].

12. SPECIFICATIONS FOR RUBBER GOODS

Directory of Specifications.—Specifications are used as a basis of purchase by many different organizations, both governmental and private. The 1,311 page National Directory of Commodity Specifications [169] prepared at the National Bureau of Standards devotes 25 pages to specifications on rubber products. Each standard or specification is listed by title, designating number, and sponsoring organization. A summary is given of the technical characteristics, scope, and special applications.

Federal Specifications.—Virtually all the rubber products used by the Federal Government from truck tires to surgeons' gloves are purchased under specifications that set standards of quality, performance, and dimensions. The savings in decreased cost and increased serviceability of the rubber products are estimated in millions of dollars. Members of the staff of the National Bureau of Standards participate in drawing up large numbers of these Federal Specifications. The general methods of physical testing and chemical analysis for rubber goods are described in Specifications ZZ–R–601a [170], which was prepared by the staff members.

13. GENERAL INFORMATION ON RUBBER

In addition to its work for other Government agencies, the National Bureau of Standards is required "to supply available information to the public, upon request, in the fields of physics, chemistry, and engineering." Individuals and small business firms constantly are asking for assistance. They are furnished information on specific topics

relating to rubber or given reference to other sources of information [174].

III. BIBLIOGRAPHY OF NBS PUBLICATIONS

The publications of the National Bureau of Standards are intended to record those phases of activity that are of general interest and permanent value. However, there are also many reports of limited interest that are submitted to other Government agencies and given circulation at the discretion of those agencies. For example, as already mentioned, more than 100 extensive mimeographed reports of work on synthetic rubber have been submitted to the Office of Rubber Reserve and circulated to participants in the national synthetic rubber program, but have not as yet been released for general distribution. Some of them have already been rewritten and issued in the form of publications, while others will probably be published later.

This section lists 174 publications, excluding unpublished reports, by members of the staff of the National Bureau of Standards under the same headings used in the preceding section. The titles of the publications show the scope of its research and technological investigations on the natural and synthetic rubbers and related materials.

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[3] "Rubber Research in Tropical Brazil." Norman Bekkedahl. India Rubber World **112**, 451 (1945).
[4] "Pa-Agronomico Method for Coagulating Rubbers." Felisberto C. de Camargo and Norman Bekkedahl. India Rubber World **109**, 473 (1944).
[5] "Some Vulcanization Tests of Guayule Rubber." David Spence and C. E. Boone. Tech. Pap. BS **22**, 1 (1927) T353.

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[16] "Effect of Pressure on the Melting of Crystalline Rubber." Lawrence A. Wood, Norman Bekkedahl, and Ralph E. Gibson. J. Research NBS **35**, 375 (1945) RP1677. J. Chem. Physics **13**, 475 (1945). Reprinted in Rubber Chem. Tech. **19**, 546 (1946).

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